FAX

Solvay Minerals Inc.
ENVIRONMENTAL DEPARTMENT

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Date December 19, 1996

Number of pages including cover sheet ~~10~~ 2

To: **Dave Luzmoor**
Solvay Minerals
c/o Kvaerner Davy

From: **Dolly Potter**
Phone **307-872-6571**
Fax Phone **307-872-6510**

Message

Dave,

Method 202 stuff, I'll talk to you at 10:00.

Dolly A. Potter

I think you are missing this page

For particulate emission testing, the Wyoming Department of Environmental Quality (WDEQ) requires utilization of Reference Method 5 sampling train, with the back half impinger catch analyzed by the protocol defined by Reference Method 202 (see attached). To determine compliance, the WDEQ will compare the sum of the Reference Method 5 front half particulate catch and the **inorganic** portion of the Reference Method 202 back half of this Method 5/202 test, against the guaranteed particulate emission rate.

petroleum and coal and is responsible for its worldwide supply, storage, and distribution.

d. Defense Industrial Supply Center (DISC). Buys and manages industrial items such as bearings, ferrous and nonferrous metals, electrical wire, gasket material, and certain mineral ores and precious metals.

e. Defense Personnel Support Center (DPSC). Buys and manages food, clothing, and medical supplies for all the armed services, some Federal agencies and authorized foreign governments.

f. Defense General Supply Center (DGSC). Buys and manages such categories of materials as electrical hardware, materials handling equipment, kitchen and laundry equipment, woodworking and metalworking machines, photographic supplies, and precision measuring instruments.

2. *Depots.* DLA depots are responsible for the receipt, storage, and distribution of DLA-managed materiel. The principal depots are:
Defense Distribution Region West (DDRW)
Defense Distribution Region East (DDRE)
Defense Depot Memphis (DDMT)
Defense Depot Ogden (DDOU)

3. *Service centers.* DLA operates six service centers which provide technical and logistics services. The service centers are:

a. Defense Logistics Services Center (DLSC). Responsible for maintenance of the Federal Supply Catalog System, including the development and dissemination of cataloging and item intelligence data to the Military Departments and other authorized customers.

b. Defense Reutilization and Marketing Service (DRMS). The central clearinghouse for the reutilization, donation, sale, or disposal of DoD-owned excess property, including scrap and waste.

c. Defense Industrial Plant Equipment Center (DIPEC). Manages the reserve of DoD-owned industrial plant equipment. The center repairs, rebuilds, and updates equipment to avoid new procurement costs.

d. DLA Administrative Support Center (DASC). Provides general administrative support to designated DLA activities.

e. Defense National Stockpile Center (DNSC). Maintains the national reserve of strategic materials stored for use in event of war or other national emergency.

f. DLA Systems Automation Center (DSAC). Develops and maintains DLA's automated and computerized systems.

4. *Contract districts.* Six districts, each responsible for contracts covering a multistate or specialized area, administer materiel contracts after they are awarded by the military services, defense agencies, some civil agencies, and certain foreign governments. The districts are:

Defense Contract Management District Northeast (DCMDN).
Defense Contract Management District Mid Atlantic (DCMDM).
Defense Contract Management District North Central (DCMDC).
Defense Contract Management District South (DCMDS).
Defense Contract Management District West (DCMDW).
Defense Contract Management Command International (DCMCI).

III. Requester Requirements

A. Addressing Requests

Address requests to the DLA PLFA most likely to hold the records (see paragraph V of this appendix for mailing addresses of FOIA managers). If the PLFA is undeterminable, address requests to HQ DLA-XAM for proper routing. Requests must be in writing.

B. Description of Records.

Provide a reasonable description of the documents you are seeking. If you have detailed information which would help reduce the search time involved, please include it in your request. If you have a document which references the DLA record you seek, include a copy of that document.

C. Fees and fee waivers.

State your willingness to pay fees above the $15 automatic waiver or provide a justification for waiver of all or part of the costs. Waiver requests must address with specificity each of the fee waiver elements in part 286, subpart F, of this title.

IV. Availability of DLA Publications

Unrestricted DLA regulations, manuals, and handbooks may be purchased from the DLA publications sales outlet. DLA Handbook 5025.1, Defense Logistics Agency Index of Publications, is published quarterly and may be used to help you identify publications of interest to you. Orders for this and other nonrestricted publications may be placed through DASC-PD, Cameron Station, Alexandria, VA 22304-6130. That office will advise you of cost before completing your order.

V. FOIA Mailing Addresses

HQ Defense Logistics Agency, Attn: HQ DLA-XAM, Cameron Station, Alexandria, VA 22304-6100.
Defense Construction Supply Center, Attn: DCSC-WXA, 3990 E. Broad Street, Columbus, OH 43216-5000.
Defense Electronics Supply Center, Attn: DESC-WXA, 1507 Wilmington Pike, Dayton, OH 45444-5252.
Defense Fuel Supply Center, Attn: DFSC-DB, Cameron Station, Alexandria, VA 22304-6160.
Defense General Supply Center, Attn: DGSC-DB, Richmond, VA 23297-5000.
Defense Industrial Supply Center, Attn: DISC-PPR, 700 Robbins Avenue, Philadelphia, PA 19111-5096.
Defense Personnel Support Center, Attn: DPSC-WXA, 2800 South 20th Street, Philadelphia, PA 19101-8419.
Defense Distribution Region East, Attn: DDRE-WX, New Cumberland, PA 17070-5001.
Defense Depot Memphis, Attn: DDMT-WX, 2163 Airways Blvd., Memphis, TN 38114-5000.
Defense Depot Ogden, Attn: DDOU-G, 800 West 12th Street, Ogden, UT 84407-5000.
Defense Distribution Region West, Attn: DDRW-WX, Tracy, California 95376-5000.
Defense National Stockpile Center Attn: DNSC-L, 1745 Jefferson Davis Highway, Crystal Square No. 4, suite 100, Arlington, VA 22202-3402.
Defense Industrial Plant Equipment Center, Attn: DIPEC-LP, 2163 Airways Blvd., Memphis, TN 38114-5051.
Defense Logistics Services Center, Attn: DLSC-WXA, 74 N. Washington Avenue, Battle Creek, MI 49017-3084.
Defense Reutilization and Marketing Service, c/o Defense Logistics Services Center, Attn: CLSC-WXA, 74 N. Washington Avenue, Battle Creek, MI 49017-3084.
DLA Systems Automation Center, Attn: DSAC-E, P.O. Box 1605, Columbus, OH 43216-5002.
DLA Administrative Support Center, Attn: DASC-RA, Cameron Station, Alexandria, VA 22304-6130.
Defense Contract Management District South, Attn: DCMDS-W, 805 Walker Street, Marietta, Georgia 30060-2789.
Defense Contract Management District Northeast, Attn: DCMDN-WX, 495 Summer Street, Boston, MA 02210-2184.
Defense Contract Management District North Central, Attn: DCMDC-WX, O'Hare International Airport, P.O. Box 66926, Chicago, Il 60666-0926.
Defense Contract Management District West, Attn: DCMDW-WXA, 222 N. Sepulveda Blvd., El Segundo, CA 90245-4320.
Defense Contract Management District Mid Atlantic, Attn: DCMDM-RW, 2800 S. 20th Street, Philadelphia, PA 19101-7478.
Defense Contract Management Command International, Attn: DCMCI-MBW, Wright-Patterson AFB, OH 45433-5000.

[FR Doc. 91-29938 Filed 12-16-91; 8:45 am]

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ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 51

[AD-FRL-3977-4]

Preparation, Adoption, and Submittal of State Implementation Plans, Method for Measurement of Condensible Particulate Emissions From Stationary Sources

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: Method 202 for the measurement of condensible particulate matter (CPM) was proposed in the Federal Register on October 12, 1990, at (55 FR 41548). This action promulgates this method. On April 17, 1990 at (55 FR 14246) EPA promulgated two methods for measuring particulate matter (PM) with an aerodynamic diameter of 10 μm or less (PM_{10}). Since CPM emissions form very fine particles in the PM_{10} size range and are considered PM_{10} emissions, the Agency is adding a method for measuring CPM emissions from stationary sources to appendix M in 40 CFR part 51. The purpose of this

rule is to provide the States with a method for measuring CPM.

EFFECTIVE DATE: December 17, 1991.

ADDRESSES: Background Information Document. The Background Information Document for the promulgated test methods may be obtained from Candace Sorrell or Peter Westlin, MD-19, U.S. EPA, Research Triangle Park, North Carolina 27711, telephone number (919) 541-1064. Please refer to "Summary of Comments and Responses for Method 202."

Docket. Docket No. A-90-03, containing materials relevant to this rulemaking, is available for public inspection and copying between 8:30 a.m. to 12 Noon and 1:30 to 3:30 p.m., Monday through Friday, at EPA's Air Docket Section, Waterside Mall, room M1500, 1st Floor, Gallery 1, 401 M Street SW., Washington, DC 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Candace Sorrell or Peter Westlin, Emission Measurement Branch (MD-19), Technical Support Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-1064.

SUPPLEMENTARY INFORMATION:

I. The Rulemaking

The EPA is proposing to add a method for measuring CPM emissions to appendix M in 40 CFR part 51 to provide a method that States can use in their State implementation plans.

II. Public Participation

The opportunity to hold a public hearing on November 2, 1990 at 10 a.m. was presented in the proposal notice, but no one desired to make an oral presentation. The public comment period was from October 12, 1990 to December 17, 1990.

III. Significant Comments and Changes to the Proposed Rulemaking

Six comment letters were received from the proposal rulemaking. A detailed discussion of these comments is contained in the background document entitled "Summary of Comments and Responses for Method 202" which is referred to in the ADDRESSES section of this preamble. The major comments raised in these letters and the Agency's responses follow.

One commenter suggests that EPA determine the chemical composition of the material collected in the sampling train to verify that it will form ambient condensibles.

The EPA believes that material will collect in the impingers only by condensation or dissolution. Dissolved gases will evaporate during analysis and will not be measured unless the gases react to form a solid or liquid while they are in solution. The EPA has designed Method 202 to prevent the formation of reaction materials from dissolved gases. The EPA believes that any remaining material collected and measured by Method 202 represents the material that would condense in the ambient air. Additional analysis of chemical composition is not necessary.

Another comment raises the concern that the method may collect some portion of the sulfur dioxide (SO_2) as condensible.

The dissolution of SO_2 in water does not lead immediately to the formation of sulfuric acid (H_2SO_4), but tends to lower the solution pH, which further inhibits sulfate or H_2SO_4 formation. The method includes a purging procedure which effectively removes SO_2 before significant oxidation occurs. No additional revisions are necessary.

The commenter feels that if EPA is allowing Method 202 to be used in conjunction with Method 201 or 201A or another dry catch procedure to determine the total PM_{10} measurement, the combined methods should be tested for precision.

The imprecision associated with combining Method 202 with Method 201 or 201A or any other dry catch procedure is not additive because each train provides a separate measurement. Since the total precision associated with the combined methods cannot be larger than the least precise sampling method, a precision evaluation of a combined sampling system is unnecessary.

A commenter suggests that EPA add specific language to the applicability section of the method stating that Method 202 cannot be used on wet sources. He notes that Method 17 is excluded from use on wet sources, and Methods 201 and 201A are not recommended for wet sources.

The EPA agrees that Method 202 with an in-stack filter is not recommended for wet sources, and such a statement has been added to the applicability. However, a heated Method 5 filter could be used in Method 202 instead of the in-stack filter which would allow application to wet sources.

One commenter requests that EPA clearly state that Method 202 should not be used for assessing compliance with emission limits set on the basis of data derived from a different measurement approach.

The EPA agrees that a violation must be shown, in the first instance, by means of measurements made with the applicable test method. Once such a showing is made, however, section 113(e) of the Clean Air Act allows the Agency to rely on any credible evidence, including evidence other than the applicable test method, to establish the duration of the period of noncompliance for the purposes of assessing a penalty.

A commenter believes that the sample collection efficiency and method precision may be affected by the sampling conditions such as impinger temperature and sampling flow rate and the method should address this possibility.

The EPA agrees that the nature of the material in the sample gas may affect collection efficiency. For example, a field demonstration of Method 202 at an oil-fired boiler resulted in about 75 percent impinger collection efficiency. This collection efficiency can be improved with the addition of a second filter place between the second and third impinger. This option has been included in the method with a discussion of applicability.

The commenter feels the 1-hour nitrogen (N_2) purge is too long. He believes the majority of the SO_2 is removed in the first few minutes. He suggests the method be revised to reduce the purge time in conjunction with maintaining the sample under cold conditions and analyzing it within 48 hours.

The EPA does not agree with reducing the purge time. Laboratory tests have shown that a 1-hour purge time is necessary to ensure the adequate removal of SO_2 from the impinger solution.

Another commenter suggests that the method should be revised to give credit for ammonium sulfate ($(NH_4)_2SO_2$) dihydrte and other condensible particulate matter formed in the gas stream due to ammonia (NH_3) injection used to enhance the efficiency of a control device.

The EPA does not agree. The condensible particulate matter formed in the gas stream due to NH_3 injection is emitted to the atmosphere. The EPA believes that condensible particulate matter emitted from the source should be counted as such even if it is a product of a pollution-control technique.

The commenter suggests that EPA consider an alternative to $MeCl_2$ consistent with the Montreal Protocol.

The EPA investigated the effectiveness of a chloroform-ether extraction during the method development phase. The chloroform-ether was not as effective as the $MeCl_2$ in removing organic materials; however, the chloroform-ether procedure was

found to be acceptable for organic extraction. The method has been revised to allow a chloroform-ether extraction.

The commenter supports the exclusion of ammonium chloride as a condensible; however, he expresses concern about $(NH_4)_2SO_4$ forming in the impingers.

The N_2 purge removes SO_2 before significant oxidation occurs. If NH_3 is present in the flue gas, the $(NH_4)_2SO_4$ formed in the impingers would not be counted as a condensible, although the H_2SO_4, which reacted with NH_3, would be counted as a condensible. Method 202 corrects for the NH_3 by measuring the sulfate using an IC analysis and subtracting out the ammonium ion (NH_4^+) mass.

The commenter agrees that the NH_3 added during the titration should be subtracted from the final weight. However, he does not agree with adding back in the water removed by the acid-base reaction.

Because H_2SO_4 is hygroscopic, the H_2SO_4 mass found in the atmosphere would have the water attached to it. The method has been revised to allow the source to correct for only the NH_4^+ or for both NH_4^+ and water as an option depending on the basis for the regulation.

A. Docket

The docket is an organized and complete file of all the information submitted to or otherwise considered by EPA in the development of this proposed rulemaking. The principle purposes of the docket are to: (1) Allow interested parties to identify and locate documents so that they can effectively participate in the rulemaking process, and (2) serve as the record in case of judicial review except for interagency review materials (Section 307(d)(7)(A)).

B. Office of Management and Budget Review

Under Executive Order 12291, EPA must judge whether a regulation is "major" and, therefore, subject to the requirement of a regulatory impact analysis. This rulemaking would not result in any of the adverse economic effects set forth in Section 1 of the Order as grounds for finding a "major rule." It will neither have an annual effect on the economy of $100 million or more, nor will it result in a major increase in costs or prices. There will be no significant adverse effects on competition, employment, investment, productivity, innovation, or on the ability of U.S.-based enterprises to compete with foreign-based enterprises in domestic or export markets. This rulemaking was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291.

C. Regulatory Flexibility Act Compliance

Pursuant to the provisions of 5 U.S.C. 605(b), I hereby certify that this attached rule, if promulgated, will not have any economic impact on small entities because no additional costs will be incurred.

This rule does not contain any information collection requirements subject to OMB review under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.*

Dated: December 6, 1991.

F. Henry Habicht II,

Acting Administrator.

List of Subjects in 40 CFR Part 51

Administrative practice and procedure,
Air pollution control,
Carbon Monoxide,
Inter-governmental relations,
Lead,
Nitrogen dioxide,
Ozone,
Particulate matter,
Reporting and recordkeeping requirements,
Sulfur Oxides,
Volatile Organic Compounds.

The EPA amends title 40, chapter I, part 51 of the Code of Federal Regulations as follows:

PART 51—[AMENDED]

1. The authority citation for part 51 continues to read as follows:

Authority: Section 110 of the Clean Air Act as amended (42 U.S.C. 7410).

2. Appendix M, to part 51 Table of Contents is amended by adding an entry to read as follows:

Method 202—Determination of Condensible Particulate Emissions From Stationary Sources

3. By adding Method 202 to Appendix M to part 51 to read as follows:

Method 202—Determination of Condensible Particulate Emissions From Stationary Sources

1. Applicability and Principle

1.1 Applicability. 1.1.1 This method applies to the determination of condensible particulate matter (CPM) emissions from stationary sources. It is intended to represent condensible matter as material that condenses after passing through a filter and as measured by this method (Note: The filter catch can be analyzed according to the appropriate method).

1.1.2 This method may be used in conjunction with Method 201 or 201A if the probes are glass-lined. Using Method 202 in conjunction with Method 201 or 201A, only the impinger train configuration and analysis is addressed by this method. The sample train operation and front end recovery and analysis shall be conducted according to Method 201 or 201A.

1.1.3 This method may also be modified to measure material that condenses at other temperatures by specifying the filter and probe temperature. A heated Method 5 out-of-stack filter may be used instead of the in-stack filter to determine condensible emissions at wet sources.

1.2 Principle. 1.2.1 The CPM is collected in the impinger portion of a Method 17 (appendix A, 40 CFR part 60) type sampling train. The impinger contents are immediately purged after the run with nitrogen (N_2) to remove dissolved sulfur dioxide (SO_2) gases from the impinger contents. The impinger solution is then extracted with methylene chloride ($MeCl_2$). The organic and aqueous fractions are then taken to dryness and the residues weighed. The total of both fractions represents the CPM.

1.2.2 The potential for low collection efficiency exist at oil-fired boilers. To improve the collection efficiency at these type of sources, an additional filter placed between the second and third impinger is recommended.

2. Precision and Interference

2.1 Precision. The precision based on method development tests at an oil-fired boiler and a catalytic cracker were 11.7 and 4.8 percent, respectively.

2.2 Interference. Ammonia. In sources that use ammonia injection as a control technique for hydrogen chloride (HCl), the ammonia interferes by reacting with HCl in the gas stream to form ammonium chloride (NH_4Cl) which would be measured as CPM. The sample may be analyzed for chloride and the equivalent amount of NH_4Cl can be subtracted from the CPM weight. However, if NH_4Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allowed to air dry at ambient temperature to prevent any NH_4Cl from vaporizing.

3. Apparatus

3.1 Sampling Train. Same as in Method 17, section 2.1, with the following exceptions noted below (see Figure 202–1). *Note:* Mention of trade names or specific products does not constitute endorsement by EPA.

3.1.1 The probe extension shall be glass-lined or Teflon.

3.1.2 Both the first and second impingers shall be of the Greenburg-Smith design with the standard tip.

3.1.3 All sampling train glassware shall be cleaned prior to the test with soap and tap water, water, and rinsed using tap water, water, acetone, and finally, $MeCl_2$. It is important to completely remove all silicone grease from areas that will be exposed to the $MeCl_2$ during sample recovery.

3.2 Sample Recovery. Same as in Method 17, section 2.2, with the following additions:

3.2.1 N_2 Purge Line. Inert tubing and fittings capable of delivering 0 to 28 liters/min of N_2 gas to the impinger train from a

standard gas cylinder (see Figure 202–2). Standard 0.95 cm (⅜-inch) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

3.2.2 Rotameter. Capable of measuring gas flow at 20 liters/min.

3.3 Analysis. The following equipment is necessary in addition to that listed in Method 17, section 2.3:

3.3.1 Separatory Funnel. Glass, 1-liter.

3.3.2 Weighing Tins. 350-ml.

3.3.3 Dry Equipment. Hot plate and oven with temperature control.

3.3.4 Pipets. 5-ml.

3.3.5 Ion Chromatograph. Same as in Method 5F, Section 2.1.6.

4. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

4.1 Sampling. Same as in Method 17, section 3.1, with the addition of deionized distilled water to conform to the American Society for Testing and Materials Specification D 1193–74, Type II and the omittance of section 3.1.4.

4.2 Sample Recovery. Same as in Method 17, section 3.2, with the following additions:

4.2.1 N_2 Gas. Zero N_2 gas at delivery pressures high enough to provide a flow of 20 liters/min for 1 hour through the sampling train.

4.2.2 Methylene Chloride, ACS grade. Blanks shall be run prior to use and only methylene chloride with low blank values (0.001 percent) shall be used.

4.2.3 Water. Same as in section 4.1.

4.3 Analysis. Same as in Method 17, section 3.3, with the following additions:

4.3.1 Methylene Chloride. Same as section 4.2.2.

4.3.2 Ammonium Hydroxide. Concentrated (14.8 M) NH_4OH.

4.3.3 Water. Same as in section 4.1.

4.3.4 Phenolphthalein. The pH indicator solution, 0.05 percent in 50 percent alcohol.

5. Procedure

5.1 Sampling. Same as in Method 17, section 4.1, with the following exceptions:

5.1.1 Place 100 ml of water in the first three impingers.

5.1.2 The use of silicone grease in train assembly is not recommended because it is very soluble in $MeCl_2$ which may result in sample contamination. Teflon tape or similar means may be used to provide leak-free connections between glassware.

5.2 Sample Recovery. Same as in Method 17, section 4.2 with the addition of a post-test N_2 purge and specific changes in handling of individual samples as described below.

5.2.1 Post-test N_2 Purge for Sources Emitting SO_2. (Note: This step is recommended, but is optional. With little or no SO_2 is present in the gas stream, i.e., the pH of the impinger solution is greater than 4.5, purging has been found to be unnecessary.) As soon as possible after the post-test leak check, detach the probe and filter from the impinger train. Leave the ice in the impinger box to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature below 20 °C. With no flow of gas through the clean purge line and fittings, attach it to the input of the impinger train (see Figure 202–2). To avoid over- or under-pressurizing the impinger array, slowly commence the N_2 gas flow through the line while simultaneously opening the meter box pump valve(s). When using the gas cylinder pressure to push the purge gas through the sample train, adjust the flow rate to 20 liters/min through the rotameter. When pulling the purge gas through the sample train using the meter box vacuum pump, set the orifice pressure differential to $\Delta H_@$ and maintain an overflow rate through the rotameter of less than 2 liters/min. This will guarantee that the N_2 delivery system is operating at greater than ambient pressure and prevents the possibility of passing ambient air (rather than N_2) through the impingers. Continue the purge under these conditions for 1 hour, checking the rotameter and ΔH value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

5.2.2 Sample Handling.

5.2.2.1 Container Nos. 1, 2, and 3. If filter catch is to be determined, as detailed in Method 17, section 4.2.

5.2.2.2 Container No. 4 (Impinger Contents). Measure the liquid in the first three impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas. Quantitatively transfer this liquid into a clean sample bottle (glass or plastic); rinse each impinger and the connecting glassware, including probe extension, twice with water, recover the rinse water, and add it to the same sample bottle. Mark the liquid level on the bottle.

5.2.2.3 Container No. 5 ($MeCl_2$ Rinse). Follow the water rinses of each impinger and the connecting glassware, including the probe extension with two rinses of $MeCl_2$; save the rinse products in a clean, glass sample jar. Mark the liquid level on the jar.

5.2.2.4 Container No. 6 (Water Blank). Once during each field test, place 500 ml of water in a separate sample container.

5.2.2.5 Container No. 7 ($MeCl_2$ Blank). Once during each field test, place in a separate glass sample jar a volume of $MeCl_2$ approximately equivalent to the volume used to conduct the $MeCl_2$ rinse of the impingers.

5.3 Analysis. Record the data required on a sheet such as the one shown in Figure 202–3. Handle each sample container as follows:

5.3.1 Container Nos. 1, 2, and 3. If filter catch is analyzed, as detailed in Method 17, section 4.3.

5.3.2 Container Nos. 4 and 5. Note the level of liquid in the containers and confirm on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container No. 4 either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Remove a 5-ml aliquot and set aside for later ion chromatographic (IC) analysis of sulfates. (Note: Do not use this aliquot to determine chlorides since the HCl will be evaporated during the first drying step; Section 8.2 details a procedure for this analysis.)

5.3.2.1 Extraction. Separate the organic fraction of the sample by adding the contents of Container No. 4 ($MeCl_2$) to the contents of Container No. 4 in a 1000-ml separatory funnel. After mixing, allow the aqueous and organic phases to fully separate, and drain off most of the organic/$MeCl_2$ phase. Then add 75 ml of $MeCl_2$ to the funnel, mix well, and drain off the lower organic phase. Repeat with another 75 ml of $MeCl_2$. This extraction should yield about 250 ml of organic extract. Each time, leave a small amount of the organic/$MeCl_2$ phase in the separatory funnel ensuring that no water is collected in the organic phase. Place the organic extract in a tared 350-ml weighing tin.

5.3.2.2 Organic Fraction Weight Determination (Organic Phase from Container Nos. 4 and 5). Evaporate the organic extract at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg.

5.3.2.3 Inorganic Fraction Weight Determination. (Note: If NH_4Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allow to air dry at ambient temperature. If multiple acid emissions are suspected, the ammonia titration procedure in section 8.1 may be preferred.) Using a hot plate, or equivalent, evaporate the aqueous phase to approximately 50 ml; then, evaporate to dryness in a 105 °C oven. Redissovle the residue in 100 ml of water. Add five drops of phenolphthalein to this solution; then, add concentrated (14.8 M) NH_4OH until the sample turns pink. Any excess NH_4OH will be evaporated during the drying step. Evaporate the sample to dryness in a 105 °C oven, desiccate the sample for 24 hours, weigh to a constant weight, and record the results to the nearest 0.1 mg. (Note: The addition of NH_4OH is recommended, but is optional when little or no SO_2 is present in the gas stream, i.e., when the pH of the impinger solution is greater than 4.5, the addition of NH_4OH is not necessary.)

5.3.2.4 Analysis of Sulfate by IC to Determine Ammonium Ion (NH_4^+) Retained in the Sample. (Note: If NH_4OH is not added, omit this step.) Determine the amount of sulfate in the aliquot taken from Container No. 4 earlier as described in Method 5F (appendix A, 40 CFR part 60). Based on the IC SO_4^{-2} analysis of the aliquot, calculate the correction factor to subtract the NH_4^+ retained in the sample and to add the combined water removed by the acid-base reaction (see section 7.2).

5.3.3 Analysis of Water and $MeCl_2$ Blanks (Container Nos. 6 and 7). Analyze these sample blanks as described above in sections 5.3.2.3 and 5.3.2.2, respectively.

5.3.4 Analysis of Acetone Blank (Container No. 8). Same as in Method 17, section 4.3.

6. Calibration

Same as in Method 17, section 5, except for the following:

6.1 IC Calibration. Same as Method 5F, section 5.

6.2 Audit Procedure. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit sample. If this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

6.3 Audit Samples. Audit Sample Availability. Audit samples will be supplied only to enforcement agencies for compliance tests. The availability of audit samples may be obtained by writing:

Source Test Audit Coordinator (MD–77B), Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U.S. Environmental Protection Agency, Research Triangle, Park, NC 27711

or by calling the Source Test Audit Coordinator (STAC) at (919) 541–7834. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

6.4 Audit Results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional Office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

7. Calculations

Same as in Method 17, section 6, with the following additions:

7.1 Nomenclature. Same as in Method 17, section 6.1 with the following additions.

C_{sms} = Concentration of the CPM in the stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

C_{SO_4} = Concentration of SO_4^{-2} in the sample, mg/ml.

m_a = Sum of the mass of the water and $MeCl_2$ blanks, mg.

m_c = Mass of the NH_4^+ added to sample to form ammonium sulfate, mg.

m_i = Mass of inorganic CPM matter, mg.

m_o = Mass of organic CPM, mg.

m_r = Mass of dried sample from inorganic fraction, mg.

V_b = Volume of aliquot taken for IC analysis, ml.

V_{ic} = Volume of impinger contents sample, ml.

7.2 Correction for NH_4^+ and H_2O. Calculate the correction factor to subtract the NH_4^+ retained in the sample based on the IC SO_4^{-2} and if desired, add the combined water removed by the acid-base reaction.

$$m_c = K\, C_{so_4}\, V_{ic} \qquad \text{Eq. 202–1}$$

where:

K = 0.0205, when correcting for NH_4^+ and H_2O.

= 0.1840, when only correcting for NH_4^+.

7.3 Mass of Inorganic CPM.

$$m_i = m_r \frac{V_{ic}}{V_{ic} - V_b} - m_c \qquad \text{Eq. 202–2}$$

7.4 Concentration of CPM.

$$C_{sms} = \frac{m_a + m_i - m_b}{Vm_{std}} \qquad \text{Eq. 202–3}$$

8. Alternative Procedures

8.1 Determination of NH_4^+ Retained in Sample by Titration.

8.1.1 An alternative procedure to determine the amount of NH_4^+ added to the inorganic fraction by titration may be used. After dissolving the inorganic residue in 100 ml of water, titrate the solution with 0.1 N NH_4OH to a pH of 7.0, as indicated by a pH meter. The 0.1 N NH_4OH is made as follows: Add 7 ml of concentrated (14.8 M) NH_4OH to 1 liter of water. Standardize against standardized 0.1 N H_2SO_4 and calculate the exact normality using a procedure parallel to that described in section 5.5 of Method 6 (appendix A, 40 CFR part 60). Alternatively, purchase 0.1 N NH_4OH that has been standardized against a National Institute of Standards and Technology reference material.

8.1.2 Calculate the concentration of SO_4^{-2} in the sample using the following equation.

$$CSO_4 = \frac{48.03\, V_t\, N}{100} \qquad \text{Eq. 202–4}$$

where

N = Normality of the NH_4OH, mg/ml.

V_t = Volume of NH_4OH titrant, ml.

48.03 = mg/meq.

100 = Volume of solution, ml.

8.1.3 Calculate the CPM as described in section 7.

8.2 Analysis of Chlorides by IC. At the conclusion of the final weighing as described in section 5.3.2.3, redissolve the inorganic fraction in 100 ml of water. Analyze an aliquot of the redissolved sample for chlorides by IC using techniques similar to those described in Method 5F for sulfates. Previous drying of the sample should have removed all HCl. Therefore, the remaining chlorides measured by IC can be assumed to be NH_4Cl, and this weight can be subtracted from the weight determined for CPM.

8.3 Air Purge to Remove SO_2 from Impinger Contents. As an alternative to the post-test N_2 purge described in section 5.2.1, the tester may opt to conduct the post-test purge with air at 20 liter/min. Note: The use of an air purge is not as effective as a N_2 purge.

8.4 Chloroform-ether Extraction. As an alternative to the methylene chloride extraction described in section 5.3.2.1, the tester may opt to conduct a chloroform-ether extraction. Note: The Chloroform-ether was not as effective as the $MeCl_2$ in removing the organics, but it was found to be an acceptable organic extractant. Chloroform and diethylether of ACS grade, with low blank values (0.001 percent), shall be used. Analysis of the chloroform and diethylether blanks shall be conducted according to Section 5.3.3 for $MeCl_2$.

8.4.1 Add the contents of Container No. 4 to a 1000-ml separatory funnel. Then add 75 ml of chloroform to the funnel, mix well, and drain off the lower organic phase. Repeat two more times with 75 ml of chloroform. Then perform three extractions with 75 ml of diethylether. This extraction should yield approximately 450 ml of organic extraction. Each time, leave a small amount of the organic/$MeCl_2$ phase in the separatory funnel ensuring that no water is collected in the organic phase.

8.4.2 Add the contents of Container No. 5 to the organic extraction. Place approximately 300 ml of the organic extract in a tared 350-ml weighing tin while storing the remaining organic extract in a sample container. As the organic extract evaporates, add the remaining extract to the weighing tin.

8.4.3 Determine the weight of the organic phase as described in Section 5.3.2.2.

8.5 Improving Collection Efficiency. If low impinger collection efficiency is suspected, the following procedure may be used.

8.5.1 Place an out-of-stock filter as described in Method 8 between the second and third impingers.

8.5.2 Recover and analyze the filter according to Method 17, Section 4.2. Include the filter holder as part of the connecting glassware and handle as described in sections 5.2.2.2 and 5.2.2.3.

8.5.3 Calculate the Concentration of CPM as follows:

$$C_{sms} = \frac{m_a + m_i + m_f - m_b}{Vm_{std}} \qquad \text{Eq. 202–5}$$

where:

m_f = amount of CPM collected on out-of-stack filter, mg.

8.6 Wet Source Testing. When testing at a wet source, use a heated out-of-stack filter as described in Method 5.

9. Bibliography

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2. DeWees, W.D. and K.C. Steinsberger. "Method Development and Evaluation of Draft Protocol for Measurement of Condensible Particulate Emissions." Draft Report. November 17, 1989.

3. Texas Air Control Board. Laboratory Division. "Determination of Particulate in Stack Gases Containing Sulfuric Acid and/or Sulfur Dioxide." *Laboratory Methods for Determination of Air Pollutants*. Modified December 3, 1976.

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6. Commonwealth of Pennsylvania, Department of Environmental Resources. Chapter 139. Sampling and Testing (Title 25, Rules and Regulations, Part I, Department of Environmental Resources, Subpart C, Protection of Natural Resources. Article III, Air Resources). January 8, 1960.

7. Wisconsin Department of Natural Resources. *Air Management Operations Handbook, Revision 3*. January 11, 1989.

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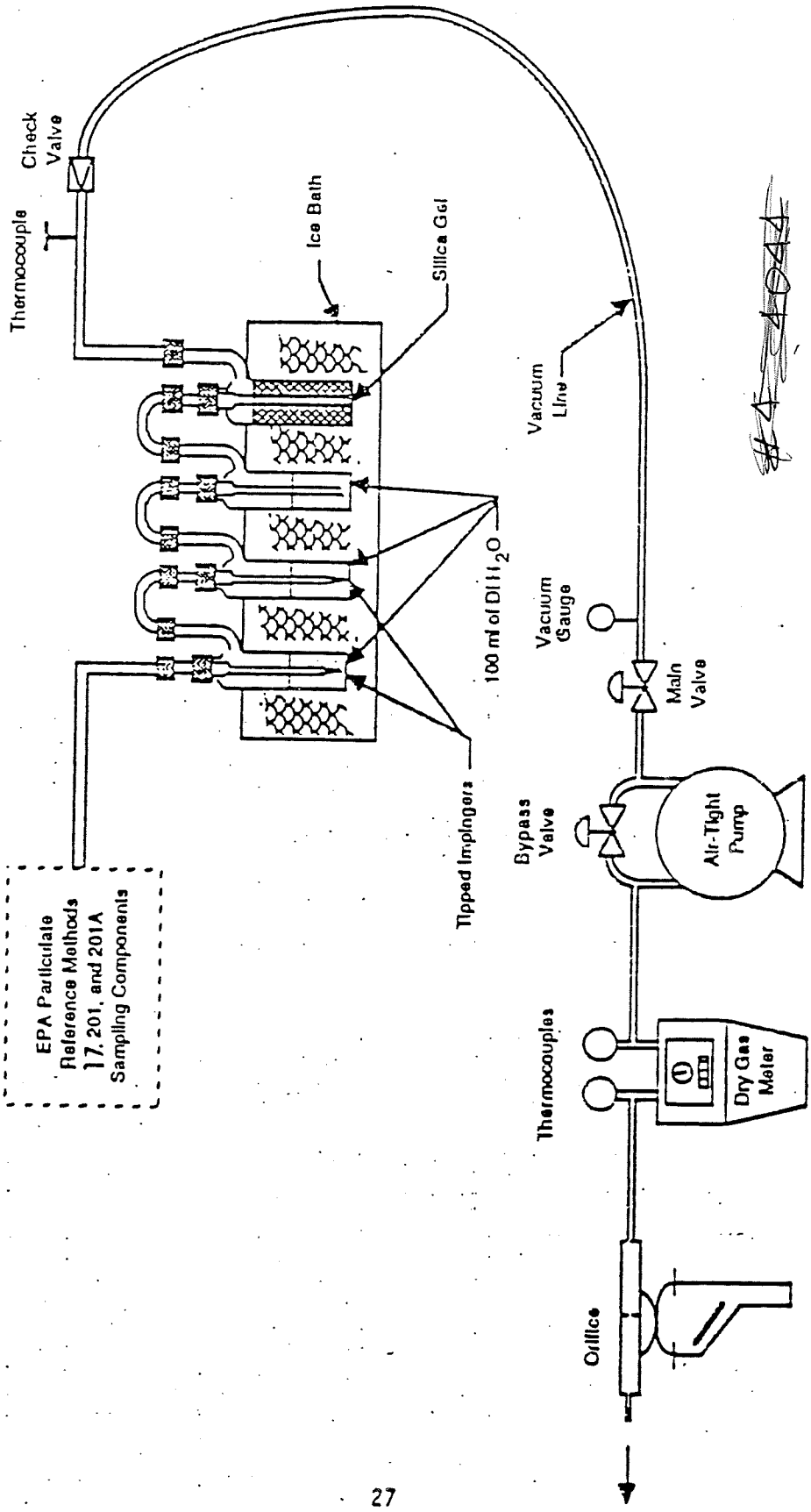

Figure 202-1. Schematic of condensible particulate sampling train.

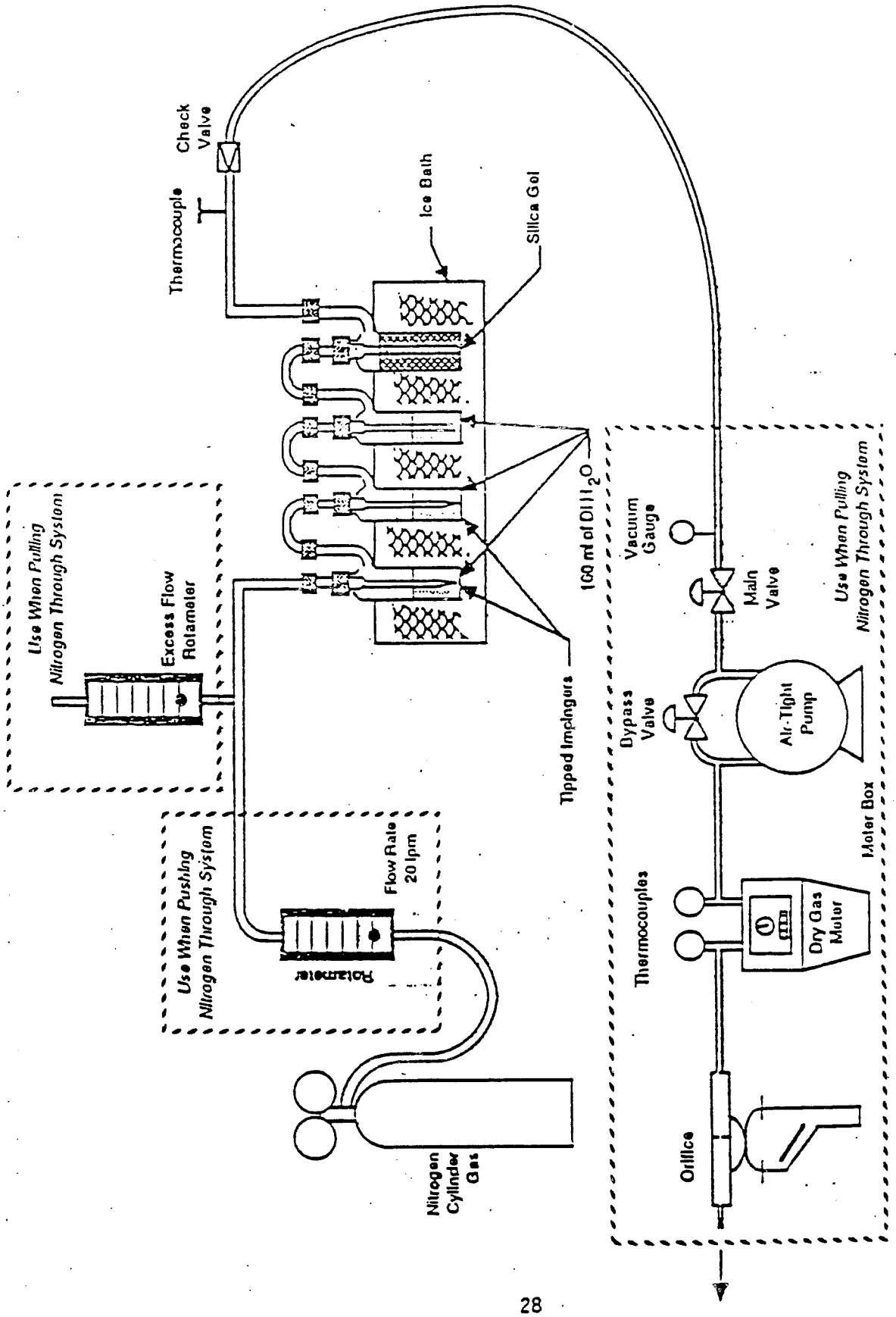

Figure 202-2. Schematic of post-test nitrogen purge system.

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Moisture Determination

Volume or weight of liquid in impingers: _____ ml or g

Weight of moisture in silica gel: _____ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport: _____ ml

Final volume: _____ ml

pH of sample prior to analysis: _____

Addition of NH_4OH required: _____

Sample extracted 2X with 75 ml $MeCl_2$?: _____

For Titration of Sulfate

Normality of NH_4OH: _____ N

Volume of sample titrated: _____ ml

Volume of titrant: _____ ml

Sample Analysis

Container number	Weight of condensible particulate, mg		
	Final weight	Tare weight	Weight gain
4 (Inorganic)			
4 & 5 (Organic)			

Total: _____

Less Blank: _____

Weight of Consensible Particulate: _____

Figure 202-3. Analytical data sheet.

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